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TITANIUM NITRIDE CERMETS

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WRIGHT AIR DEVELOPMENT CENTER

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TITANIUM NITRIDE CERMETS

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July 1952

*Flight Research Laboratory
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**Wright Air Development Center
Air Research and Development Command
United States Air Force
Wright-Patterson Air Force Base, Ohio**

FOREWORD

This report was prepared by F. K. Davey, E. R. Glabau and G. E. Lorey at the New Jersey Ceramic Research Station, Rutgers University, under the supervision of Dr. John H. Koenig, Director, and Dr. R. B. Sosman, Faculty Adviser. Presented herein are the results of laboratory experiments concerned with the evaluation of titanium nitride-base cermets. This work was accomplished for the U. S. Air Force under the provisions of Contract No. W33-038-ac-15800. The technical phases of the contract were administered by the Flight Research Laboratory, Directorate of Research, Wright Air Development Center, with Mr. Murray A. Schwartz acting as project engineer under RDO 463-7, "High Temperature Materials."

ABSTRACT

Several compositions of cermets based on titanium nitride were investigated. The cermets were tested for strength in cross bending, resistance to oxidation, and resistance to thermal shock. The differences of the various compositions are noted as well as the effect of different processing techniques.

Titanium nitride cermets containing chromium as the metal phase were found to have the best properties of the compositions investigated.

PUBLICATION REVIEW

This report has been reviewed and is approved.

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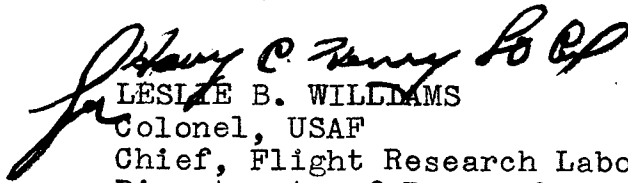

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TITANIUM NITRIDE CERMETS

Introduction

The desire for higher operating temperatures in jet type engines for aircraft has brought with it the demand for materials which will give satisfactory service in such engines at temperatures above 1800°F. Such materials must resist severe thermal shock and have good strength at the operating temperatures. They must have resistance to the corrosive effects arising from the various fuels and atmospheres and withstand the erosive effect of the hot high velocity gases. Efforts to develop materials suitable for use in jet and rocket engines have led to considerable research on refractory metals, silicates, oxides, graphite, combinations of metal with oxides, and combinations of metal with metallic interstitial compounds. The borides, carbides, nitrides, and silicides have many interesting properties and many are being investigated as possible refractories for jet engines.

Titanium nitride is an interstitial compound of titanium and nitrogen in 1:1 molar ratio, though the phase is stable from $\text{TiN}_{1.0}$ to $\text{TiN}_{0.42}$. The material having a Ti:N ratio of 1:1 has a density of 5.21 and melts at 3220°C.¹

Titanium nitride is quite stable having a decomposition pressure of about 10^{-3} atmospheres at 2200°C.² It is stable in the presence of carbon to about 1300°C above which temperature titanium carbide will form. Titanium nitride is not stable in the presence of oxygen and oxides will form with appreciable velocity at temperatures above 500°C, converting the nitride to TiO and

subsequently to Ti_2O_3 and TiO_2 . The nitride will form solid solutions with both TiC and TiO . The rate at which oxidizing reactions proceed is an important property of cermets.

Titanium nitride crystallizes in the $NaCl$ structure with a lattice parameter of 4.235 A. for $TiN_{1.0}$ decreasing to 4.213 for $TiN_{0.42}$.

The work of Ueltz³ with mixtures of MgO , NiO , and TiN indicated that cermets of good strength may be developed using TiN as the base.

Various mixtures of TiN and metal were made and treated in different ways in order to evaluate the effect of both the methods of processing and the composition on the properties of the cermets. Variations in the methods of grinding, mixing, and firing produced greater variations in the strength of the finished test bars than did changes of composition, though it will be shown later that the changes in grinding time made very significant changes in the composition of the cermet.

Experimental Details

The TiN used was purchased from the Metal Hydrides Co., Beverly, Mass. A typical analysis* of this material shows it to have a nitrogen to titanium ratio of 0.9. X-ray patterns showed the $NaCl$ structure with a lattice parameter of 4.23Kx. The metals used were technical grade -325 mesh supplied by Charles Hardy Co., New York.

*Analysis courtesy of the Titanium Division, National Lead Co., South Amboy, New Jersey.

All grinding was performed in sample size hardened steel ball mills. The mills were charged with 200 g. of the material to be ground, 100 cc methyl alcohol, and 1000 grams of 5/8" steel balls. When the results showed the large amount of iron picked up from the grinding, tungsten carbide slugs were used instead of steel balls.

The length of time the materials were milled was a significant variable and produced large changes of strength.

After milling, the material was dried, granulated to pass an 80 mesh sieve and pressed hydrostatically into bars 5/8" x 5/8" x 4 1/2"; these bars were then cut into bars approximately 1/4" x 1/4" x 2".

The bars were then fired in an induction furnace using a graphite sleeve as a susceptor. An atmosphere of dried nitrogen was maintained during the firing. The firing schedule used in all cases was: 1 1/2 hours to temperature, hold for 1/2 hour, cool to 2300°F in 1/2 hour. The firing temperature used was either 2700°F, 2900°F, 3000°F, or 3200°F.

Some bars from each mixture were used to determine the modulus of rupture after firing. Other bars were used to measure the resistance to oxidation.

The procedure used to test the bars for resistance to oxidation was as follows: The bars were first weighed and measured. They were then put into an electric furnace maintained at 2000°F for eight hours in an atmosphere of air. After being removed from the furnace, the bars were cooled, weighed, and then broken in a cross

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bending test. The data obtained were the weight gained, and the strength after oxidation.

The porosity was determined using at least one sample from each batch.

Selected compositions were tested for thermal shock.

Bodies Composed of Titanium Nitride

A study was first made of the influence of the grinding procedure on the TiN. The standard mill charge was used (Steel balls) and the grinding time was varied. The times used were 4, 8, 24, 48, 72, and 96 hours. Bars were prepared and fired at 3200°F. The results of this are given in Table I and Figure 1.

It should be noted here that in addition to grinding time, another variable, inherent to the procedure, was introduced - that of iron pickup from the steel ball mill and balls during grinding. Therefore, the values reported are not those of TiN but of a TiN-Fe series with the amount of iron increasing as the time of grinding increased.

The modulus of rupture greatly increased with grinding time, reaching 50,800 psi for the 96 hour grind, while the porosity decreased to 0.77%. The per cent weight gain on oxidation was practically the same for all grinds; the decrease in porosity with increasing grinding time evidently was offset by the greater iron pickup for the longer grinding times, and thus there was no noticeable increase in oxidation resistance for the denser bodies. The strength after oxidation for all grinds was poor.

X-ray diffraction patterns of all samples after firing gave the typical TiN pattern. No iron peaks were evident for the shorter grinding periods, but as the iron pickup increased, these Fe peaks were detected for the 72 and 96 hour grinds. All the oxidized samples revealed rutile and ilmenite (FeTiO_3), the peaks of the latter becoming more intense as the time of grinding increased. Alpha Ti_2O_3 was detected in the 96 hour grind.

Since the 96 hour grind had the best properties to date, it was analyzed to determine the iron content. A gravimetric " R_2O_3 " procedure was used. Some titanium, which will come down with the R_2O_3 group, was also removed during the HCl digestion, the amount being dependent upon the time of digestion. The TiN as received was analyzed along with the 96 hour grind samples so the amount of titanium removed would be known and the true iron content could be calculated. For the 96 hour grind, 14.0% iron was present due to pickup from the grinding operation.

In an attempt to obtain data on pure TiN, the material ground 96 hours was leached with HCl to remove the iron. A series of digestions, centrifugings (to retain as much as possible of the colloidal TiN), and washing was used. The "leached" TiN was then tested in the same manner as the TiN of the grinding series. The properties of this material are listed in Table I.

The strength of this leached TiN - 17,300 psi - was much less than that of the 96 hour grind with the iron present. This would indicate that the iron is a beneficial addition for increasing the strength of a TiN body. However, it is possible that the leaching

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action of the HCl may have changed the character of the TiN enough so that a low strength value was obtained.

The excellent strength and low porosity of the 96 hour grind, with iron present, was somewhat offset by the low oxidation resistance and strength after oxidation. When tungsten carbide slugs had been procured, some test bars were formed from material that had been ground for 96 hours with these slugs substituted for the steel balls. The bars were tested in the usual manner and the results are also given in Table I. The high strength of these bars indicates that the low strength of the acid leached material is due in large part to an effect other than the absence of iron.

Titanium Nitride-Nickel Cermets

A total grinding time of 96 hours was chosen for each TiN-Ni series. In order to study the effect of milling the metal and TiN together for different times with the same total grinding time for the TiN, the TiN was milled alone for a certain period and the metal was then added to the mill to complete the 96 hour grind. Thus, for each composition, the Ni and TiN were milled together for 24, 48, 72, or 96 hours. In Table II the notations of grinding time indicate the time of milling the metal and TiN together based on a total 96 hour grind for the TiN.

The standard charge in the mill was used. Additions of 5, 10, 20, 30, and 35% Ni were tried for each grinding time until the Ni "sweated" out on firing. Test bars were prepared and fired in a nitrogen atmosphere at 2700° and 2900°F.

After firing, the room temperature strength, porosity, oxidation resistance, and strength after oxidation values were obtained.

Table II presents the data for the TiN-Ni series. The strength values were substantially increased over that of the 96 hour grind TiN fired at 3200°F, and the oxidation resistance was somewhat improved by the nickel metal additions. It was felt that the poor oxidation resistance of these TiN cermets was due to the high iron content. The strength was reduced about 50% after 8 hours oxidation at 2000°F for all compositions.

Figures 2 and 3 present the room temperature modulus of rupture values versus the time of milling the TiN and Ni together for each composition and temperature. It can be seen that some compositions reached peak strength and then fell off, some are leveling off, and for others the strength is steadily increasing. For example: the 5% Ni fired at 2700° or 2900°F reached its highest strength value at 72 hours of mixing and then fell off, while the 10% Ni fired at 2900°F is still increasing in strength.

The lower metal contents - 5% for the 24, 48, and 72 hour grinds, and 5 and 10% for 96 hours - gave the highest values for each series. It should be remembered, however, that approximately 14% iron was picked up during the grinding; therefore, these nominally low metal content samples actually contain 20 to 25% metal. As was expected, increased time of milling the metal and TiN together increased the amount of metal retained by the TiN.

Polished sections indicated excellent metal distribution for all samples, the metal being the discontinuous phase. With increased time of milling the metal and TiN together, the individual metal "pools" became smaller and more numerous.

Another series of TiN-Ni mixtures were made and were ground using tungsten carbide slugs in place of steel balls. Spectrographic analysis showed the mixtures had picked up 3% iron and 0.1% WC. Bars were formed, fired, and tested in the usual way. The results are given in Table III and Figure 4.

It can be seen from Figure 4 that as the per cent Ni metal was increased, the strength both after firing and after oxidation decreased with the exception of the 20% Ni body. The maximum strength after firing of this TiN-Ni series was obtained for the 20% Ni addition. The strength after oxidation is extremely low. The highest per cent Ni addition (25% Ni) not only gives the lowest values for the strength after firing (2700° and 2900°F) but also the lowest strength values after oxidation for this series.

By comparison with the values in Table III for 100% TiN ground 96 hours with tungsten carbide and fired at 3200°F it is noticed that none of the Ni additions appreciably improved the properties of the pure TiN.

Comparing the results in Table II, (metal grinding time - 96 hours) with the results in Table III, (15, 20, and 25% Ni additions), it can be seen that the reduction in iron pickup during grinding did not appreciably lower the per cent weight gain in oxidation. It is also noticeable that the strength after firing

was reduced substantially by decreasing the iron pickup; the strength after oxidation was decreased slightly.

Titanium Nitride-Cobalt Cermets

Mixtures of TiN and cobalt were prepared using tungsten carbide slugs for grinding. Bars were formed, fired and tested in the usual way. The results are given in Table III and Figure 5.

Figure 5 indicates a continuous increase of strength after firing and after oxidation (2900°F) and suggests that an increase in the amount of Co added is needed in order to reach the optimum strength conditions. From the results in Table III, it is evident that the proper firing temperature is 2900°F or more for the Co additions. The strength of these cermets after oxidation is very low and the weight gained during oxidation is very high.

Titanium Nitride-Chromium Cermets

Mixtures of TiN and chromium were prepared using tungsten carbide slugs for grinding. Bars were formed, fired, and tested in the usual way. Results are given in Table III and Figure 6.

The TiN-Cr cermets show the greatest promise of the three TiN-metal series investigated. An increase in the amount of Cr added to TiN increased the strength both after firing and after oxidation as well as decreased the amount of weight gained during oxidation. From Figure 6 and the results in Table III, it can be seen that for a still greater addition of chromium, the strength and oxidation resistance might increase.

The TiN-Cr series exhibited good oxidation resistance; although the strength after firing was mediocre, no significant change in strength was found after oxidation.

From the foregoing results of the investigation, it was decided to study cermet bodies containing chromium and nickel or cobalt added to titanium nitride. The TiN-Co bodies had good strength values while the TiN-Cr bodies exhibited good oxidation resistance. It was hoped that a combination of Cr with Ni or with Co would give a TiN cermet with both good strength and good resistance to oxidation.

Mixtures were made of TiN-Ni-Cr, and TiN-Co-Cr, and tungsten carbide slugs were used for grinding. The total metal additions for each of the two series were 10, 15, 20, and 25%; in each case, the metal addition was a 1:1 weight mixture either of Ni and Cr or of Co and Cr. The firing temperatures were 2700° and 2900°F in a nitrogen atmosphere. The bars were tested in the usual way. Some compositions were tested for resistance to thermal shock. The results are presented in Table IV and Figures 7 and 8.

Titanium Nitride-Nickel-Chromium Cermets

From Figure 7, it can be seen that 2900°F was the better firing temperature. The 20% metal addition gave the highest strength after oxidation while the 15% addition showed the least loss of strength after oxidation. None of the TiN-Ni-Cr bodies had better strength after firing than TiN alone; the weight gained during oxidation was not appreciably improved; but the strength after oxidation was better than that of TiN alone.

Titanium Nitride-Cobalt-Chromium Cermets

The 10% metal addition (2900°) gave the maximum strength for this series, and although the strength was reduced one-third after oxidation, this body was considerably better than TiN alone. The 25% metal addition (2700°) showed the least loss of strength after oxidation.

Thermal Shock Tests

The resistance to thermal shock of four compositions, selected from the foregoing work on the basis of strength before and after oxidation, was tested. For this a separate set of bars were prepared. The materials were ground with tungsten carbide slugs, and the bars were fired to 2900°F. The testing was accomplished as follows: The bars were mounted in an insulating brick base and inserted in a furnace maintained at 2000°F. After 15 minutes, the samples were withdrawn and set in an air blast (77 ft/sec.) for at least 5 minutes. The bars were given twenty or forty cycles and were then broken in cross bending. The loss of strength due to this treatment is a measure of the ability of the material to withstand thermal shock.

The compositions used and the results of this test are given in Table V and Figure 9. Since in the course of this test the bars are subjected to high temperatures for a considerable length of time and loss of strength due to oxidation will also take place. The strength of these compositions after oxidation is given in Table V for comparison.

The results suggest the following significant facts:

1. The 20% Cr, 80% TiN and 10% Co-Cr, 90% TiN compositions show no significant loss of strength due to this thermal shocking. It may be that the test is not severe enough to be significant.

2. The Ni-Cr-TiN compositions show a large loss of strength due to this treatment.

It will be noted in examining Table V that the original strength of the bars of the 10% Co-Cr, 90% TiN is not as high as the strength found in the first set of bars of this composition which is given in Table IV. The reason for this change in strength of bars from one batch to another has not been determined. A comparison of Table IV and Table V shows that only this composition changed in this way.

Summary

Several TiN-metal compositions were prepared and tested for strength, resistance to oxidation, and resistance to thermal shock. The effect of compositional changes and of changes in the grinding and mixing procedure are noted.

The highest strength found in any of these cermets was 73,000 lbs/in² in cross bending. This strength was exhibited by two compositions, 25% Co, 75% TiN, and a body having a nominal composition of 10% Ni, 90% TiN which had a true composition of 9% Ni, 14% Fe, 77% TiN due to iron pickup during mixing and grinding.

The strength of both of these compositions dropped to 21,000 lbs/in² after being oxidized for eight hours at 2000°F.

The highest strength found in these cermets after eight hours oxidation at 2000°F was 39,800 lbs/in² exhibited by the 20% Cr, 80% TiN composition. This is slightly higher than the strength of the unoxidized cermet.

After the tests for thermal shock the strength of the cermet of composition 20% Cr, 80% TiN had not been lowered significantly. The strength of the TiN-Ni cermets had decreased markedly. The TiN-Co cermets tested showed a slight decrease in strength.

The TiN-Cr cermets appear to have the best properties for use in jet engines. The strength of these cermets is only moderately high, but the strength was not lost during the oxidation or thermal shock tests conducted. The data of Table III and Figure 6 indicate that higher strengths may be obtained by changes in the amount of metal used in the cermet and by changing other conditions of processing.

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Table I
Properties of TiN Fired to 3200°F in Nitrogen

Grinding Time Hours	M.O.R. after Firing psi	Apparent Porosity %	Wt. Gain on Oxidation %	M.O.R. after Oxidation %
0	4,560	38.1	11.23	6,480
4	11,550	34.3	12.14	7,350
8	17,600	28.7	12.99	8,000
16	18,700	20.0	11.53	4,490
24	21,800	17.7	11.65	4,860
48	31,200	5.7	12.26	6,270
72	43,900	1.2	13.03	10,480
96	50,800	0.8	11.54	16,100
96-Fe Free	17,300	2.0	12.61	4,610
96	45,100	0.6	5.95	20,600

¹TiN as received (-325 mesh)

²96 hour grind leached with HCl

³Ground with tungsten carbide slugs

Table II
Properties of TiN - Ni Mixtures
Ground with Steel Balls

Metal Grinding Time - Hours	% Ni	Firing Temp. °F	M.O.R. after Firing - psi	Apparent Porosity - %	Wt. Gain in Oxidation - %	M.O.R. after Oxidation - psi
24	5	2700	59,100	0.1	6.54	34,450
		2900	45,200	0.2	6.64	24,000
	10	2700	52,800	0.2	6.24	20,800
		2900	44,800	0.4	6.32	19,600
	20	2700 ¹	39,800		6.28 ²	14,600
		2900 ¹	36,100		6.92	20,000
48	5	2700	68,600	0.2	5.60	31,300
		2900	53,800	0.2	6.03	27,800
	10	2700	47,900			
		2900	57,500	0.4	7.95 ³	22,400
	20	2700	39,100	0.5	7.67	17,000
		2900	50,000	1.2		
72	30	2700 ¹	32,400			
		2900 ¹	31,000			
	5	2700	71,600	0.3	6.62	33,650
		2900	58,800	0.3	6.64	21,600
	10	2700	66,800		6.70	19,600
		2900	65,100		6.81	16,800
96	20	2700	63,100		6.49	15,300
		2900	51,000			
	30	2700 ¹	50,800	0.8	11.54	16,100
	0	3200 ³	45,100	0.6	5.95	20,600
		2700	59,150	0.3	5.47	33,500
	5	2900	67,300	0.5	7.06	22,000
	10	2700	67,050	0.1	6.99	18,500
		2900	73,300	0.4	6.82	20,900
	20	2700	59,800		6.34 ²	18,600
		2900	56,300		6.52 ²	21,200
	30	2700	63,400		--- ²	29,600
		2900	62,000		5.59 ²	38,900
	35	2700 ¹	30,900	6.5		

¹ Metal sweated out on firing

² Oxide layer spalled off

³ Ground with tungsten carbide slugs

Table III

Properties of TiN Cermet

% Metal	Firing Temperature	M.O.R. After Firing	% Apparent Porosity	% Wt. Gain in Oxidation	M.O.R. After Oxidation
TiN	3200	45,100	0.6	5.95	20,600
15 Ni	2700	48,000	0.3	5.41	-----
	2900	46,200		4.92	23,300
20 Ni	2700	24,450		6.79	12,200
	2900	49,350		6.68	20,100
25 Ni	2700	23,725		----- ²	11,700
	2900	34,485		7.78	16,600
15 Co	2700	29,550	< 0.1	9.51	15,600
	2900	67,500		9.52	18,600
20 Co	2700	47,125	< 0.1	6.28	19,100
	2900	70,775		8.40	20,000
25 Co	2700	54,375	0.1	6.84	18,200
	2900	72,725		7.87	21,200
15 Cr	2900	34,075	8.4	2.21	28,500
	3000	31,175		1.98	31,900
20 Cr	2900	37,725	1.4	0.88	39,800
	3000	43,250		0.98	39,400

Note: All samples ground with tungsten slugs

¹ Warped on firing² Oxide layer flaked off

Table IV
Properties of Tin Cermets

% Metal ³	Firing Temp., °F	M.O.R. After Firing, psi	% Apparent Porosity	% Wt. Gain on Oxidation	M.O.R. after Oxidation, psi
0	3200	45,100	0.6	5.95	20,600
10 Ni-Cr	2700 2900	29,800 39,000		7.04 7.11	20,700 22,700
15 Ni-Cr	2700 2900	31,300 44,100	--- ¹ 7.4	4.91 4.76	28,800 28,100
20 Ni-Cr	2700 2900	41,700 38,100	--- ¹ ---	4.26 4.08	33,000 33,000
25 Ni-Cr	2700 2900	23,800 38,300	--- ¹ ---	3.78 ² 3.16 ²	20,700 32,200
10 Co-Cr	2700 2900	50,900	< 0.1 < 0.1	6.60 6.66	30,100 35,500
15 Co-Cr	2700 2900	42,100 34,800	< 0.1 ---	7.40 7.77	23,800 23,100
20 Co-Cr	2700 2900	35,200 37,200	12.3 10.5	6.52 6.69	23,400 22,100
25 Co-Cr	2700 2900	34,600 44,000	10.2 8.3	3.94 5.45	28,600 23,400

¹ Samples cracked on firing

² Oxide layer flaked off

³ Metal mixture contains equal weights of Ni and Cr or Co and Cr

Table V
Thermal Shock Properties of TiN Cermets

<u>Composition</u>	<u>Initial Strength</u>	<u>Strength After Thermal Shock</u>		<u>Strength after Oxidation</u>
		20 cycles	40 cycles	
10 Co-Cr	40,200	40,700	38,400	35,500
15 Ni-Cr	43,900	37,300	37,200	28,100
20 Ni-Cr	36,000	27,000	27,600	33,000
20 Cr	37,000	36,900	35,600	39,800

Effect of Grinding on Strength of TiN Steel Balls

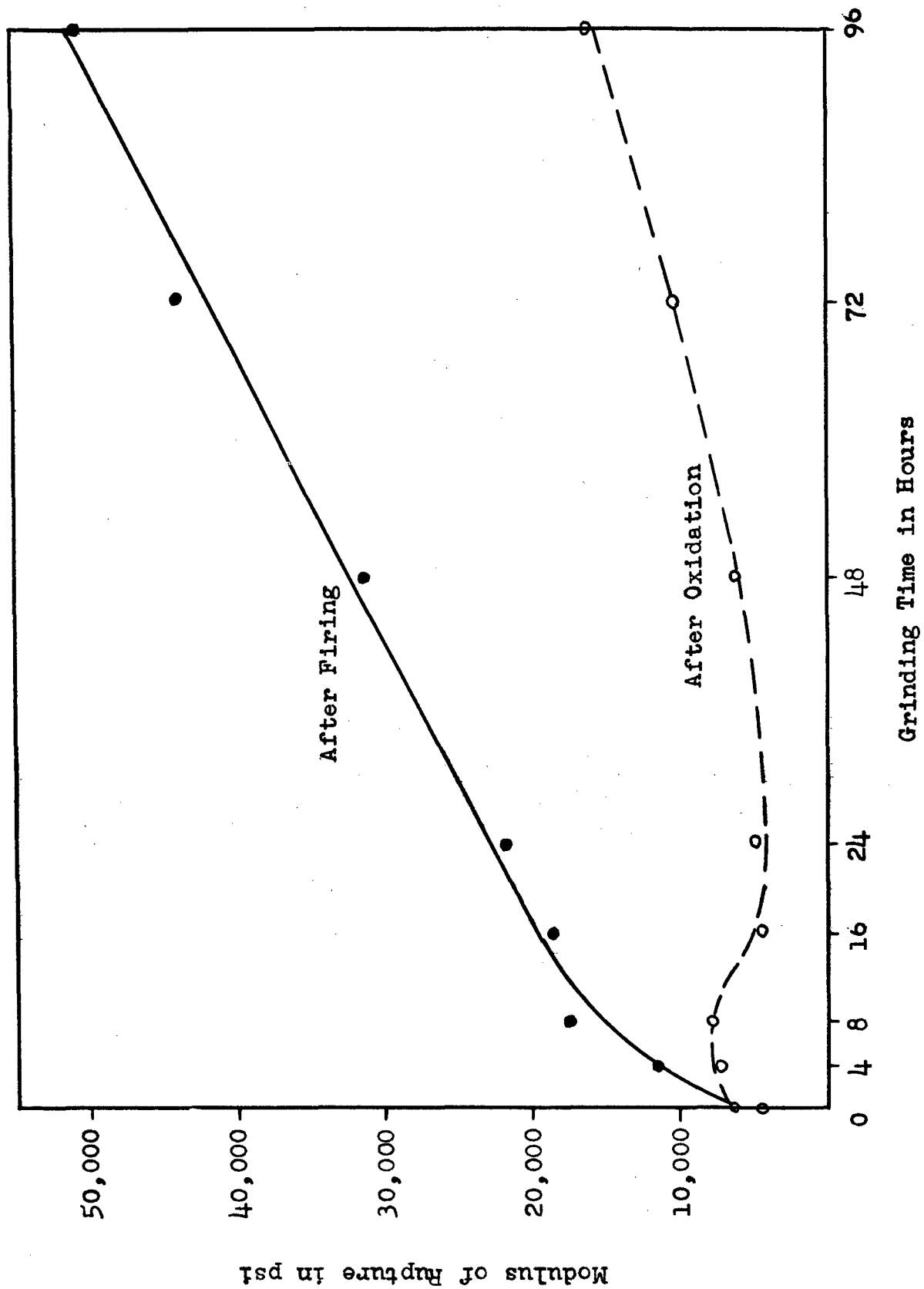
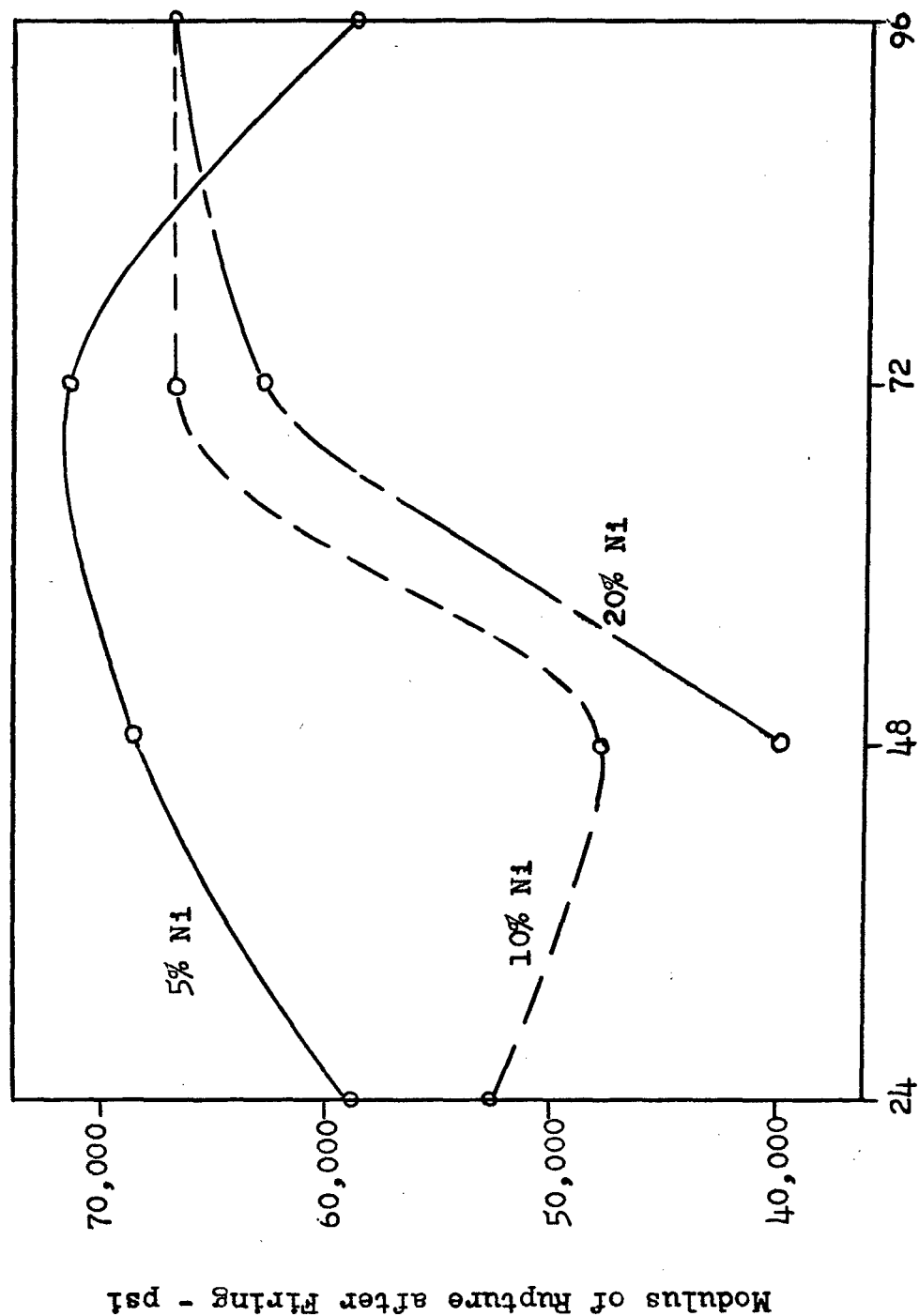


Figure 1

TiN-Ni Cermet Fired at 2700°F
Total 96 Hour Grind - Steel Balls



Time in Hours - Ni Milled with TiN

Figure 2

TiN-Ni Cermets Fired at 2900°F
Total 96 Hour Grind - Steel Balls

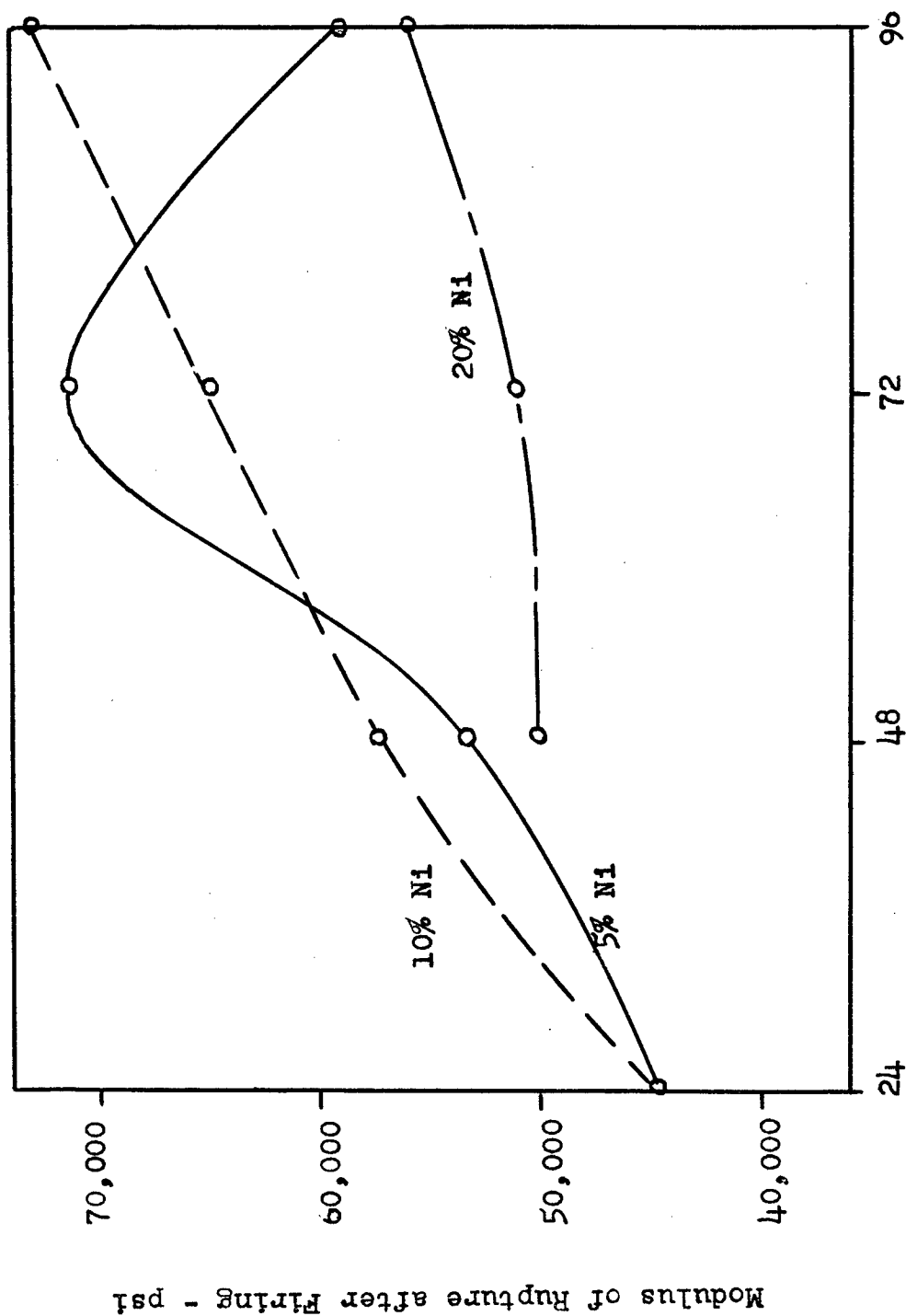


Figure 3
Time in Hours - Ni Milled with TiN

TiN-Ni Cermets Fired at 2700°F and 2900°F

Total 96 Hour Grind with WC Slugs

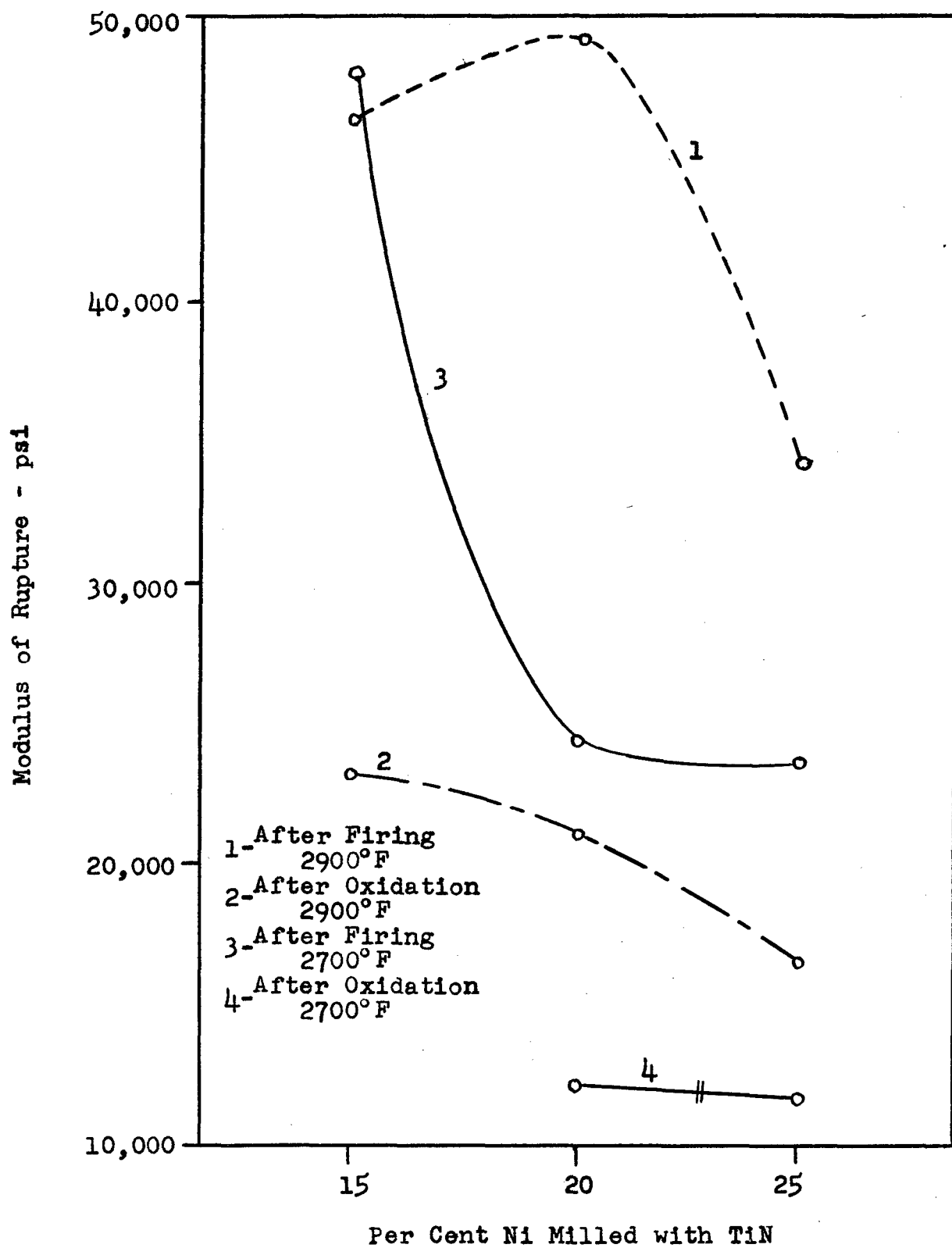


Figure 4

TiN-Co Cermets Fired at 2700°F and 2900°F

Total 96 Hour Grind with WC Slugs

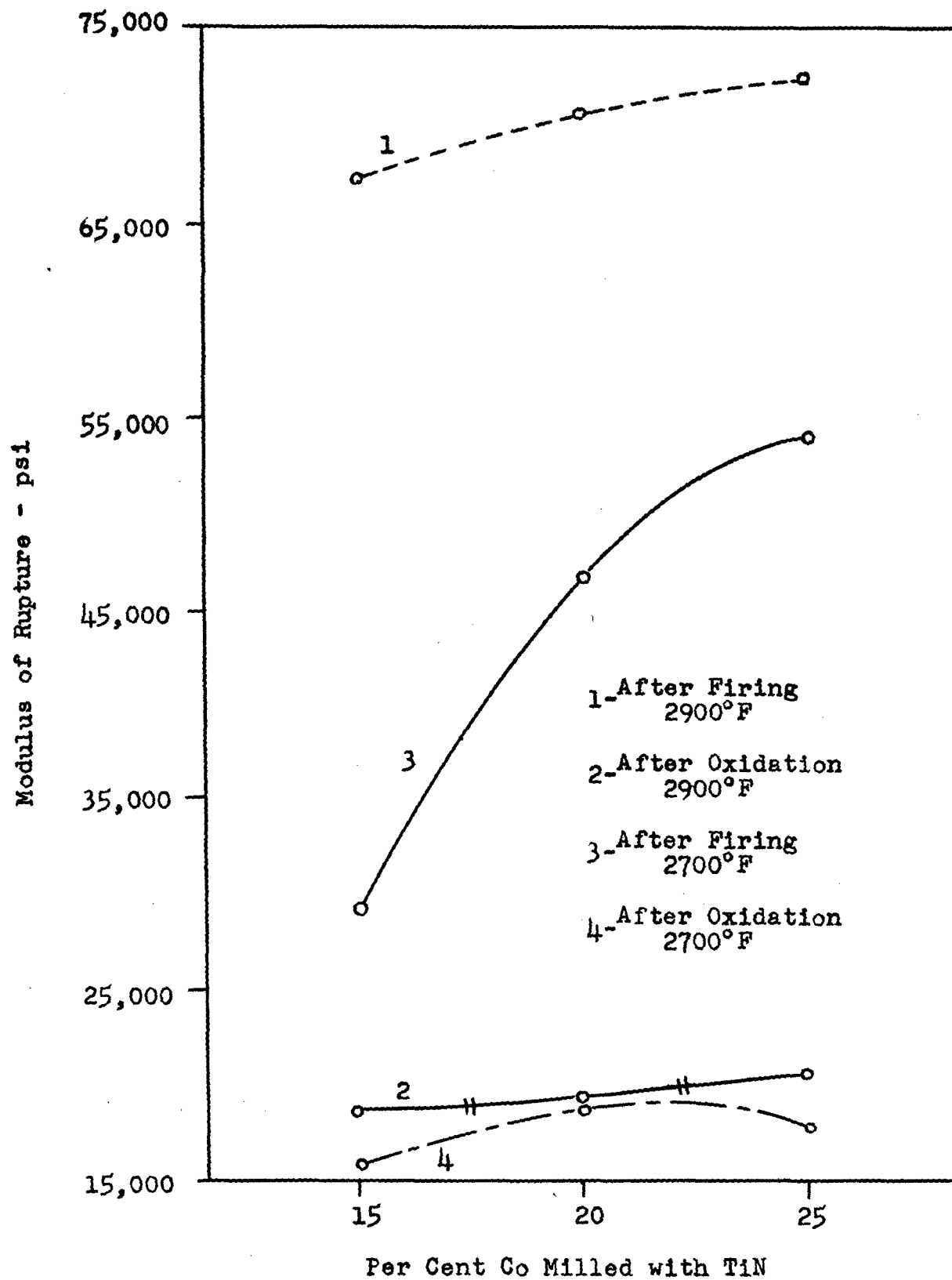


Figure 5

TiN-Cr Cermets Fired at 2900°F and 3000°F

Total 96 Hour Grind with WC Slugs

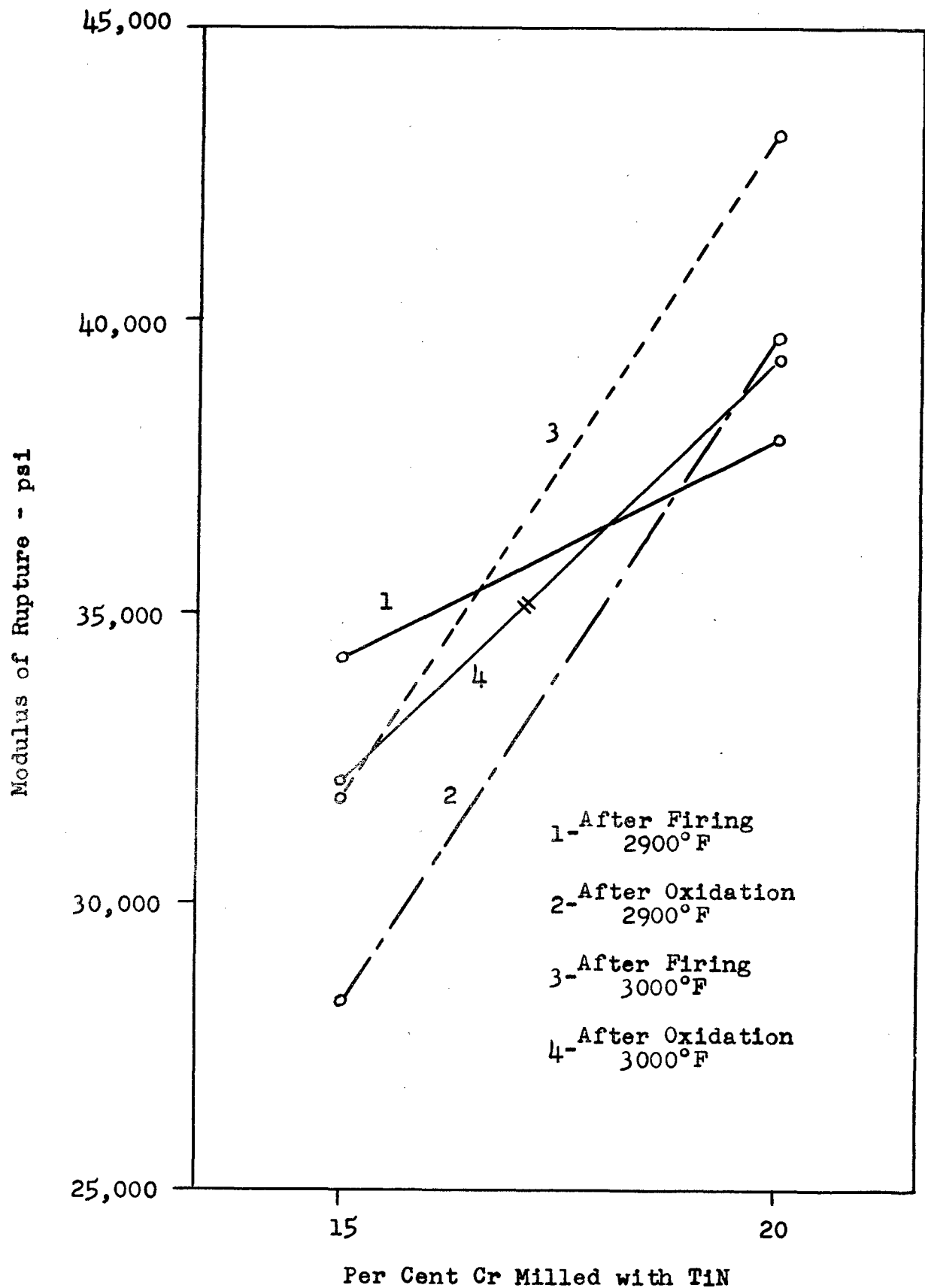
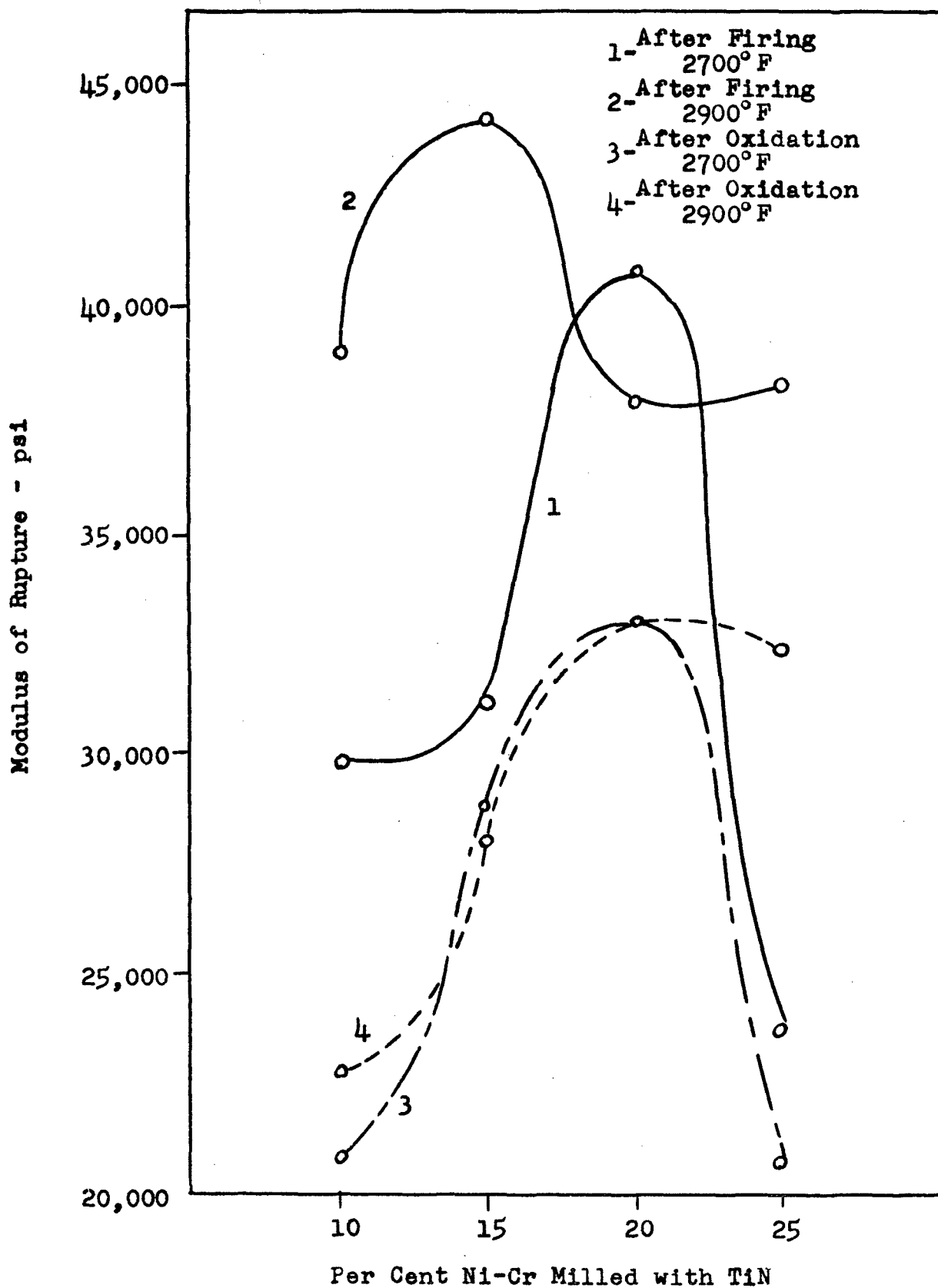


Figure 6

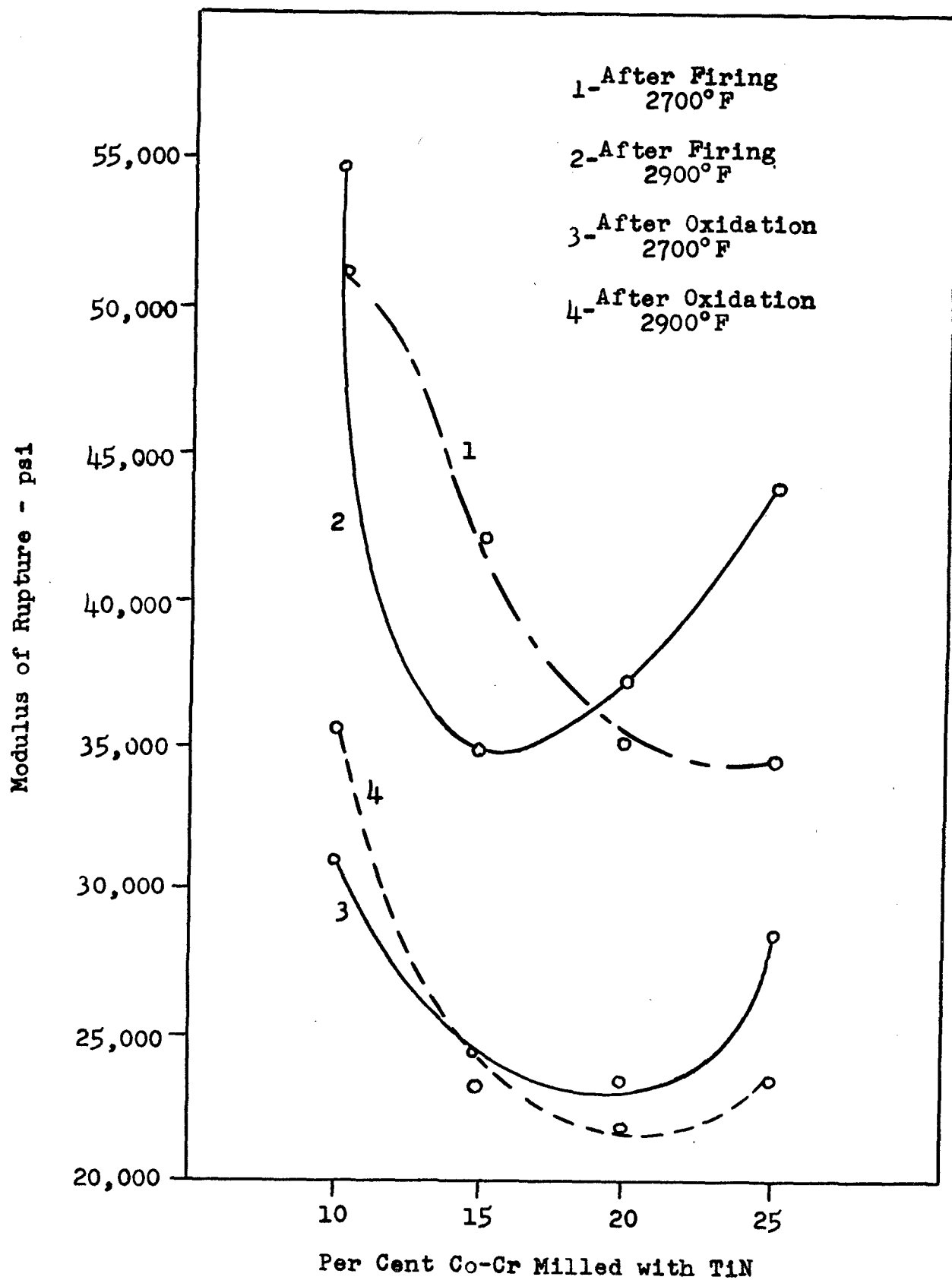
TiN-Ni-Cr Cermets Fired at 2700°F and 2900°F

Total 96 Hour Grind with WC Slugs



TiN-Co-Cr Cermets Fired at 2700°F and 2900°F

Total 96 Hour Grind with WC Slugs



Thermal Shock Properties
of TiN Cermets

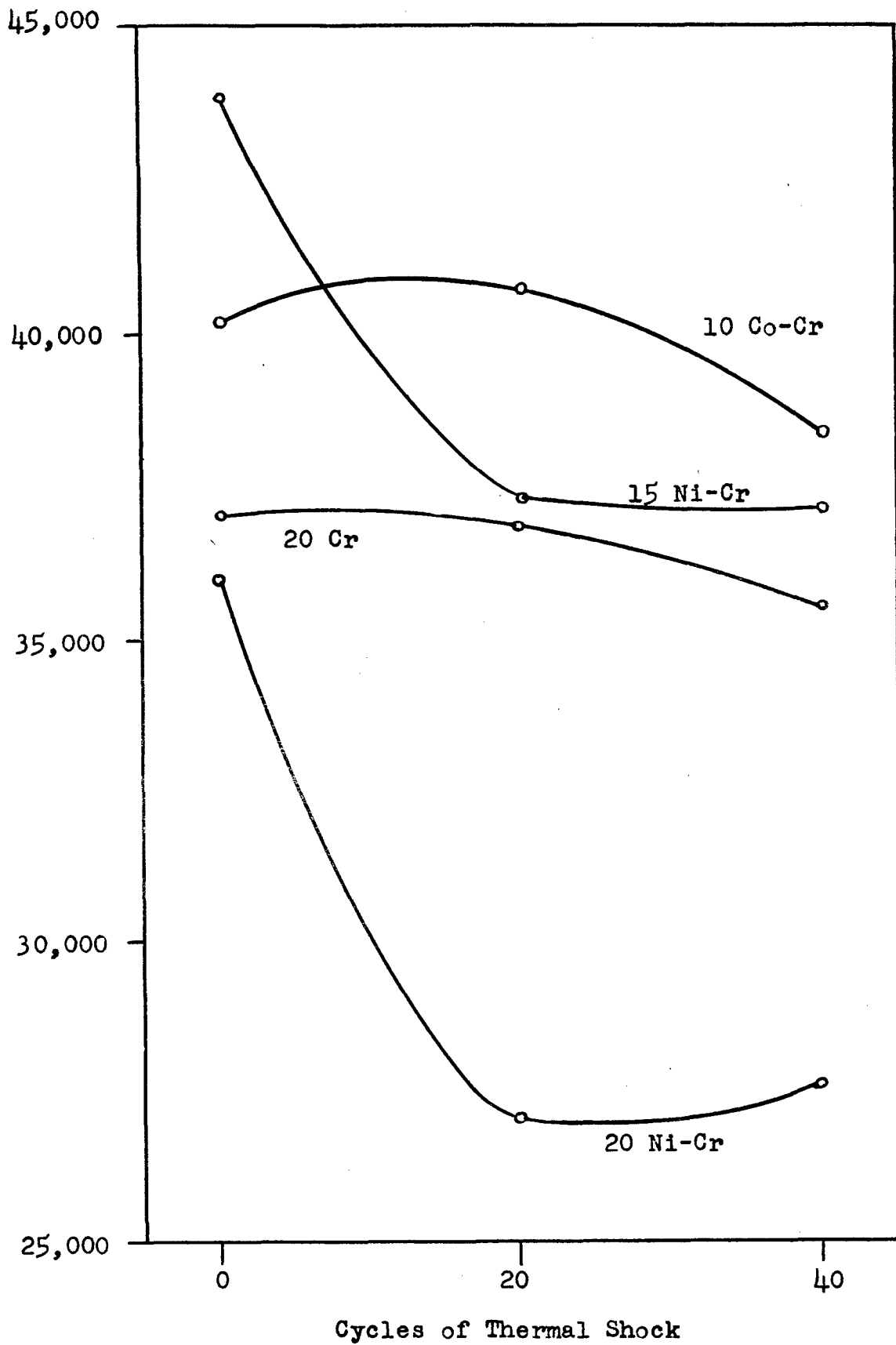


Figure 9